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EFFECT OF ULTRAVIOLET IRRADIATION ON SELECTED PLASTIC FILMS IN VACUUM

by Evelyn Anagnostou
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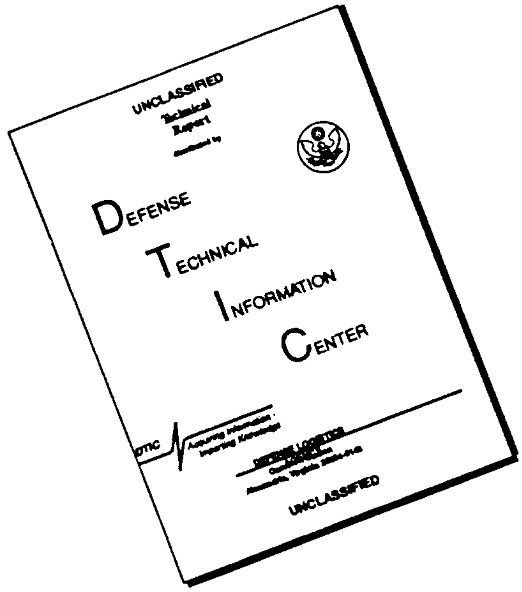

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SUMMARY

Selected plastic films which had possible application as encapsulants for thin-film cadmium sulfide solar cells were irradiated in vacuum by ultraviolet light below 3000 angstroms for various lengths of time up to 5266 hours. The light intensity was between 0.67 and 1 times the integrated solar intensity below 3000 angstroms at 1 astronomical unit. The change in optical transmission with irradiation time was measured from 0.35 to 2.7 microns, and changes in percent elongation and breaking strength were measured at room temperature. Of the films tested, the most resistant to damage was H-film, a polyimide, and weather-durable Mylar was the next most resistant.

INTRODUCTION

Reaction to the space environment for a wide range of materials has been the object of much effort in recent years. Plastic films may be useful in the space environment in various applications, depending on how rapidly the films are damaged by this environment. One possible use of these films is to encapsulate thin-film cadmium sulfide solar cells, which might be used as sources of power in space. For this type of solar cell, the plastic film provides several functions: protection from the prelaunch environment, physical support, and a method of temperature control in space.

When a plastic film is used as an encapsulant, the effects of the space environment on the optical transmission and the mechanical properties are important. The films must remain reasonably transparent (80 to 90 percent) to most of the solar spectrum of interest and should maintain good mechanical properties for a period of at least 2500 hours.

Only a small amount of work evaluating the effects of electromagnetic radiation on plastic films (refs. 1 to 6) has been done with ultraviolet light in the 1000- to 3000-angstrom range in vacuum. Mainly, the interest has been in irradiating Mylar, Teflon, nylon, and polyethylene. The light sources used have been varied, and the intensities have been either unspecified (refs. 1 to 4), 4 to 4.5 suns from 2500 to 4000 angstroms (ref. 5), or 6.5 suns from 2700 to 4000 angstroms (ref. 6). Some of these experiments have been merely qualitative.

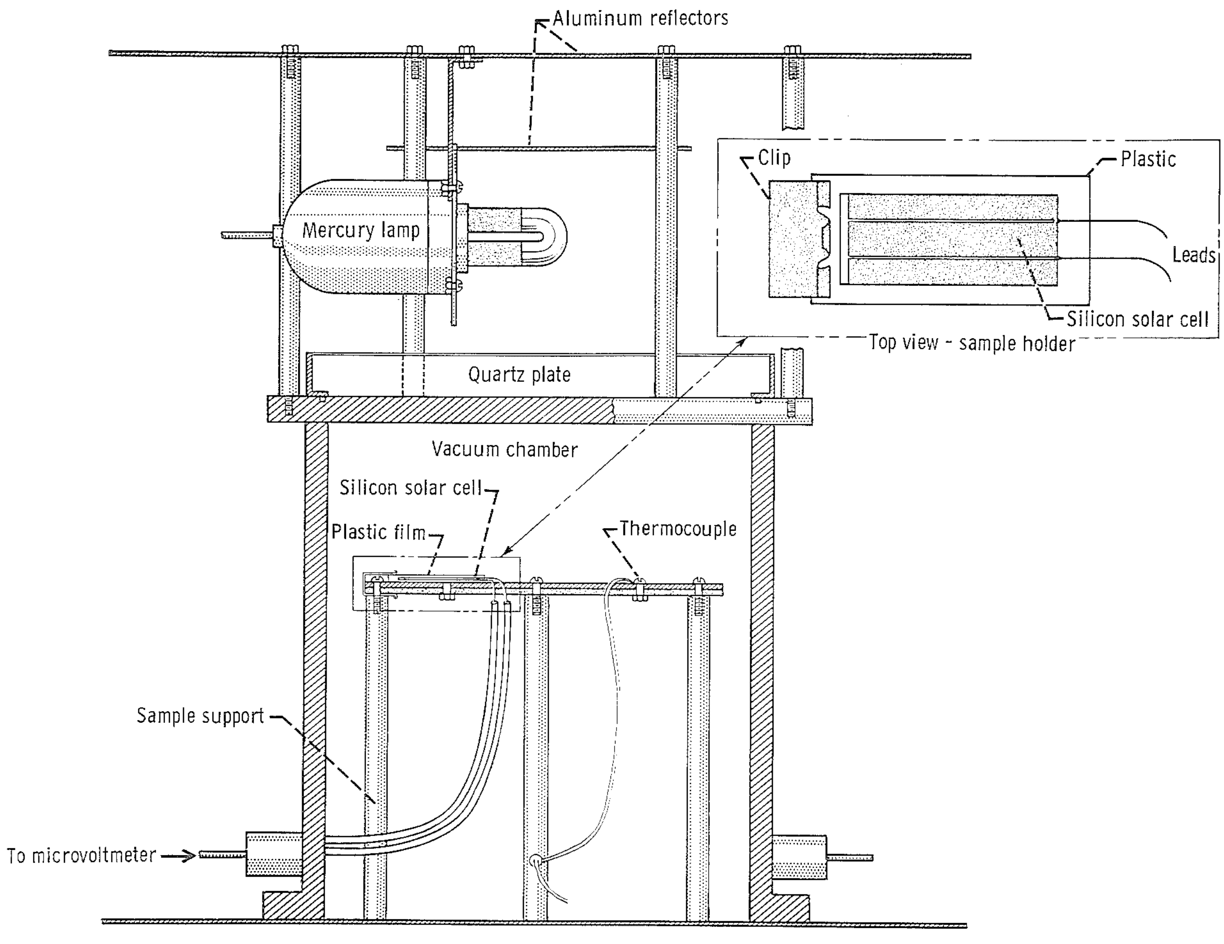

Figure 1. - Apparatus for short-term ultraviolet irradiation test.

TABLE I. - PLASTIC FILMS TESTED

Trade name	Chemical composition	Manufacturer	Thickness, in.
Aclar 22 A	Fluorohalocarbon	Allied Chemical Corp.	0.005
Visotherm	Polyethylene	Union Carbide Corp.	.0015
Weather-durable Mylar	Polyethylene terephthalate plus 100 EXP 341A	E. I. Du Pont de Nemours and Co.	.001
Phenoxy-8	Polyhydroxy ether	Union Carbide Corp.	.001
Udel X-1	Polypropylene	Union Carbide Corp.	.001
Trithene A	Polychlorotrifluoro ethylene	Union Carbide Corp.	.001
Mylar plus adhesive	Polyethylene terephthalate plus GT 300	E. I. Du Pont de Nemours and Co.	.001
Mylar	Polyethylene terephthalate	E. I. Du Pont de Nemours and Co.	.001
Tedlar 20	Polyvinylfluoride	E. I. Du Pont de Nemours and Co.	.001
Tedlar 30	Polyvinylfluoride	E. I. Du Pont de Nemours and Co.	.001
Capran	Polyamide	Allied Chemical Corp.	.0005
H-Film	Polyimide	E. I. Du Pont de Nemours and Co.	0.001, 0.0005
Kynar	Poly (vinylidene fluoride)	Penn Salt Chemicals Corp.	.001
-----	Poly (2,6 dimethyl phenylene ether)	NASA Langley Research Center	.001
H-film prepolymer	Polyimide cast from Pyre ML varnish in dimethyl formamide	NASA Langley Research Center	.0005

In this study, the effect of ultraviolet irradiation on selected plastic films in vacuum was studied by the measurement of the changes produced in the optical transmission and the mechanical properties (percent elongation and breaking strength).

The transmission of the films was measured in the range 0.35 to 2.7 microns, and the mechanical properties were measured at room temperature. Initially, many films were available for testing. Primary screening was accomplished with a short-term (1003 hr) exposure to ultraviolet light in vacuum; in addition, the films were irradiated with electrons. The intensity of the ultraviolet light was equal to the integrated solar intensity between 2000 and 3000 angstroms at 1 astronomical unit. In addition, the films were irradiated with 2-million-electron-volt electrons for a total dose of 5×10^{16} electrons per square centimeter. The three films that performed best were tested further by irradiation with ultraviolet light in a long-term test (up to

5266 hr). The intensity of this light was equal to two-thirds of integrated solar intensity below 3000 angstroms at 1 astronomical unit. Supplemental tests were also performed on various coatings and special plastic formulations.

APPARATUS AND PROCEDURE

Screening Tests

A sketch of the experimental apparatus used for the first screening tests is shown in figure 1. Small samples (1 by 1 in.) of plastic film were mounted over silicon cells around the periphery of a metal plate in a vacuum chamber. The average pressure during the irradiation was 3×10^{-6} torr, and the average temperature, measured by a thermocouple on a metal plate, was 48° C.

The plastics were irradiated through a 1-inch-thick quartz window, with the light from a Hanovia 100-watt, type SH, high-pressure, quartz mercury vapor lamp, placed 9.6 inches from the samples. The intensity of the mercury lamp was equivalent to that of the Sun in the 2000- to 3000-angstrom region at 1 astronomical unit. The lamp intensity was measured with the system uranyl sulfate - oxalic acid as an actinometer (ref. 7). Also included in the test chamber as controls were a bare silicon cell and a silicon cell covered with a 1/8-inch-thick piece of Pyrex.

Qualitative measurements of the transmission of the films at various time intervals were made by measuring the response of the silicon cells at various wavelengths with filters and light from a 500-watt General Electric photoflood lamp, PH/RFL2. The filters were Farrand blocked interference filters with peak wavelengths at 0.392, 0.435, 0.498, 0.547, 0.610, 0.698, 0.803, and 0.899 micron. At the end of the test (1003 hr) the mechanical properties (percent elongation and breaking strength) of both unirradiated and irradiated films were measured with an Instron Model TT-C tensile tester equipped with rubber-faced jaws. The initial separation of the jaws was 1/2 inch in all cases. Pulling rates of 0.1, 0.2, and 0.5 inch per minute were employed. This is one of the areas where the test procedure does not so conform with the recommendation of the ASTM (ref. 8) that the results obtained should not be compared with standard values. The samples ranged from 1/4 to 1/2 inch in width and from 1 to 2 inches in length and were cut without regard to direction. The transmission spectra were measured on a Perkin-Elmer Model 350 spectrophotometer in the wavelength range 0.35 to 2.7 microns.

Initially, 12 plastics were studied. These plastics are the first 12 listed in table I.

An intermediate short-term test was performed on Aclar 22A, Udel X-1, Mylar, and weather-durable Mylar. The plastics were covered with silicon monoxide coatings ranging from 1200 to 1700 angstroms in thickness. By this means it was hoped that the ultraviolet light would be absorbed in the coating and that the film would thereby be protected from ultraviolet damage. The test was made in the same apparatus used for the early tests except that two mercury lamps were used. The coated films were affected as much as the uncoated films were.

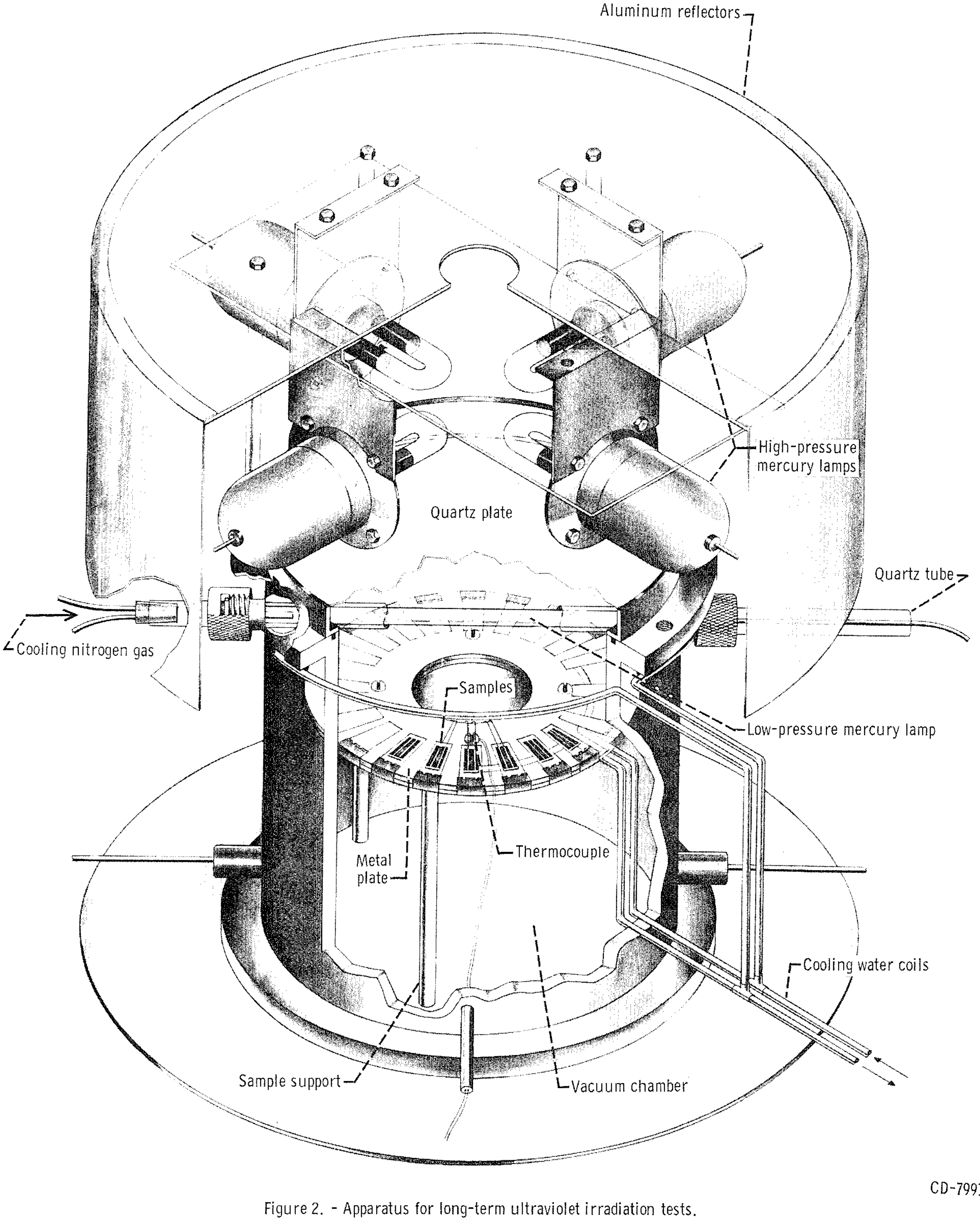

Figure 2. - Apparatus for long-term ultraviolet irradiation tests.

TABLE II. - COMPARISON OF MECHANICAL AND OPTICAL PROPERTIES OF FILMS

[Test conditions: total time, 1003 hr; average temperature, 48° C; average pressure, 3×10^{-6} torr; intensity, equal to integrated solar intensity below wavelengths of 300 mμ.]

Plastic	Elongation, percent (a)		Tensile strength at break, psi (b)		Original transmission wavelenth, percent		
	Unirradiated	Irradiated	Unirradiated	Irradiated	350 mμ	500 mμ	800 mμ
Aclar 22A	410	110	6×10^3	3×10^3	83	94	98
Visotherm	>1200	4	5	5	58	77	90
Weather-durable Mylar	120	(c)	24	10	(d)	84	98
Phenoxy-8	60	1	8.3	5	20	80	98
Udel X-1	93	9	27	8	65	91	98
Trithene A	550	2	7	3	72	88	98
Mylar plus GT300 adhesive	110	28	10	7	8	43	69
Mylar	69	3	13	10	18	84	98
Tedlar 20	130	42	17	12	14	53	94
Tedlar 30	270	54	11	8	10	53	96
Capran	340	8	6	12	25	75	97
1-Mil H-film	110	78	21	20	(e)	95	100

[a]Error: ±10 for percent >10, ±1 for percent <10.

[b]Error: $\pm2\times10^3$.

[c]Sample too brittle to test.

[d]Completely absorbing below 350 mμ.

[e]Completely absorbing below 450 mμ.

Long-Term-Tests

In the first series of long-term tests, three of the plastic films that had been tested earlier, H-film, weather-durable Mylar, and Tedlar 30, were chosen for further study. These tests were conducted to determine whether prolonged irradiation would confirm early results. Also, since shorter wavelength ultraviolet radiation would be expected to affect the plastic film more readily, some shorter wavelength radiation was included as part of the source. A sketch of the apparatus is shown in figure 2. Four of the 100-watt mercury arc lamps previously described were used (11.5 in. from the samples) in addition to a special 2-watt low-pressure mercury lamp made by Hanovia, which has 2 percent of its output at 1849 angstroms and 86 percent of its output at

2537 angstroms. This lamp was enclosed in a thin-walled quartz tube, transparent at 1849 angstroms, and cooled with nitrogen gas. This tube was situated between the samples and the quartz window, about 2.75 inches from the samples. The samples were placed on a metal plate that was water cooled. The average temperature of the plate was 42.5° C, and the pressure was in the range 0.5×10^{-6} to 2×10^{-6} torr. Several samples of each plastic were irradiated; after a certain period of irradiation, one of each type was removed and tested. The mechanical properties were measured on the Instron unit mentioned earlier. The transmission was measured with the Perkin-Elmer Model 350 spectrophotometer in the wavelength range 0.35 to 0.75 micron. The tests on the Tedlar 30 film were discontinued after 1071 hours, since electron damage tests on cells showed severe degradation (private communication), and the transmission of the films in the blue end of the spectrum also decreased rapidly. The other films were tested for a total of 5266 hours at a light intensity equivalent to two-thirds of the integrated solar intensity below 3000 angstroms at 1 astronomical unit.

In the second series of long-term tests, the following new materials were tested in the same chamber:

(1) Plastic films

   (a) Poly (dimethylphenylene ether)

   (b) Kynar, a poly (vinylidene fluoride)

   (c) H-film prepolymer, which had been cast from Pyre M-L varnish (DuPont) in dimethyl formamide

(2) A clear silicone potting compound, General Electric LTV 602, which had been successfully used in zinc oxide paint formulations for space work

(3) A composite of a prepolymerized H-film cast on weather-durable Mylar

The three plastic films are also listed in table I (p. 3).

The silicone was applied on a glass slide primed with the recommended General Electric primer S-4044. A second slide was coated with primer only to check whether any effects observed were due to the primer.

Four samples of the H-film prepolymer were tested; one film was as cast, and the other three were cured at times and temperatures recommended for the Pyre M-L varnish. These latter samples will be referred to as H_1, cured for 2 minutes at 400° C; H_2, cured for 100 minutes at 200° C; and H_3, cured for 16 hours at 150° C.

These films were tested similarly to those discussed in the previous paragraph except that, since only one sample of each was available, only the transmission was periodically checked. The mechanical properties were measured on the original and final samples only. Also, no mechanical property measurements could be made on the silicone primer and paint since they were painted on glass slides.

RESULTS AND DISCUSSION

Screening Tests

The results of the early tests are shown in table II. Included are comparisons of the mechanical properties of the unirradiated and the irradiated samples and the percent of the original transmission at three wavelengths for the irradiated samples. The wavelengths chosen were 350, 500, and 800 millimicrons, representative values for the near ultraviolet, visible, and near infrared regions. Spectra were also compared in the infrared region, but no changes could be detected. All the films but one showed very little transmission loss at 800 millimicrons. Many had not darkened appreciably at 500 millimicrons, which is close to the peak of solar intensity. Almost all the films darkened appreciably at 350 millimicrons, except Aclar 22A, Udel X-1, and Trithene A. Weather-durable Mylar is coated with a substance that acts as an ultraviolet absorber, and thus the film is completely absorbing below 350 millimicrons. H-film, because of its structure, becomes completely absorbing below 450 millimicrons. These five films were considered promising with respect to transmission stability. The change in response with time of the silicon cells covered with the plastic films was qualitatively similar. The response decreased most at the low wavelengths and very slightly (if at all) at the high wavelengths. This decrease was evidenced visually by the films becoming from slightly tan to very brown in color. These results agree with those reported previously in the literature for nylon and polyethylene (refs. 2 and 3), Mylar (refs. 2, 3, 5, and 6), polyvinyl fluoride (ref. 5), and polyamide (ref. 5), which indicated various amounts of film darkening with ultraviolet irradiation. Comparing the mechanical properties in table II shows that, in most cases, the strength at breaking went down. Most films became brittle under irradiation, as evidenced by the large drop in percent elongation and flexibility. Again these results are similar to those reported previously for some of the films, which indicated in general a decrease in elongation and tensile strength at breaking and lessened flexibility (refs. 1 and 5) with ultraviolet irradiation.

Since brittleness is the more important of these properties, insofar as solar cells are concerned, the films that still had good percent elongation after irradiation were considered worthy of further study. These were Aclar 22A, Tedlar 20, Tedlar 30, and H-film. All the films which seemed good with respect to either transmission stability or percent elongation after ultraviolet irradiation were subsequently irradiated with 2-million-electron-volt electrons for 10 to 15 minutes for a total dose of 5×10^{16} electrons per square centimeter. There was no apparent change for both Tedlar films, weather-durable Mylar, H-film, and regular Mylar, while samples of Udel X-1, Aclar 22A, and Trithene A became brittle with this dose (private communication).

When these results were considered, weather-durable Mylar, Tedlar 30, and H-film were irradiated in a long-term test.

Long-Term Tests

<u>First series</u>. - The change in transmission with irradiation of Tedlar 30,

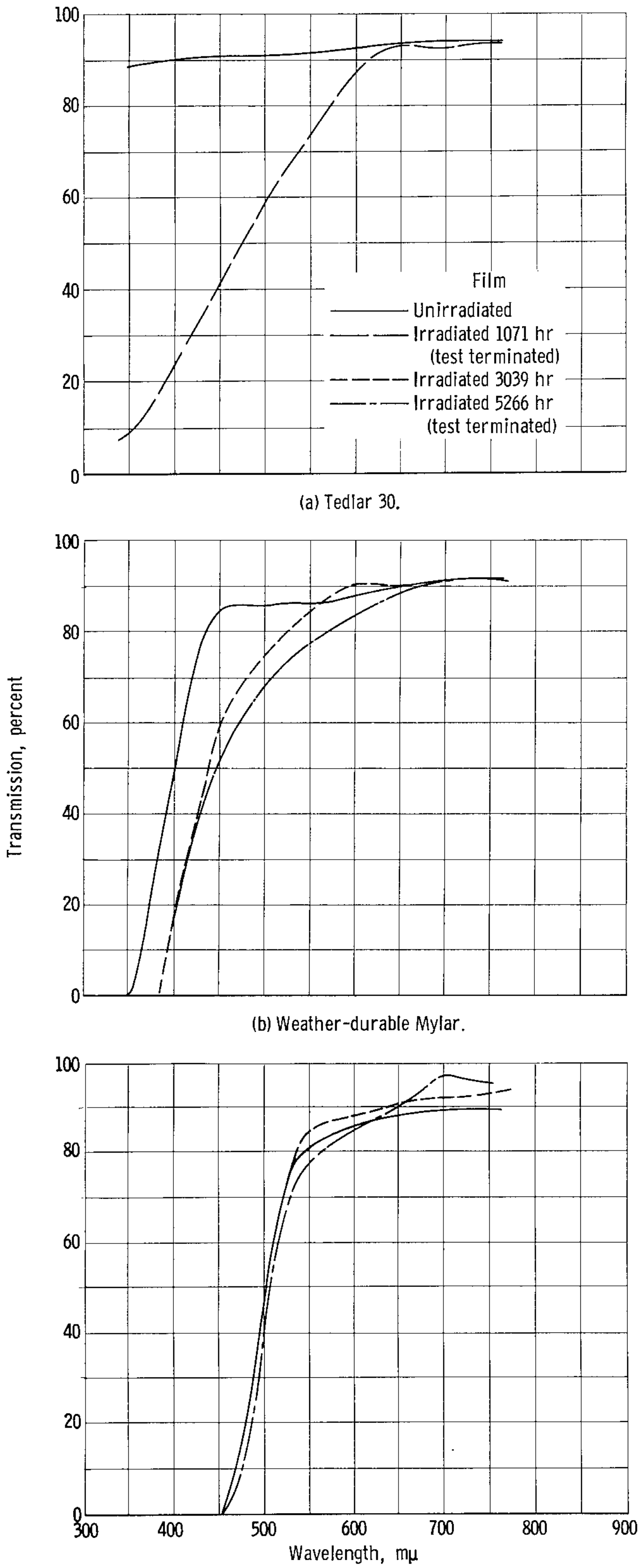

Figure 3. - Effect of long-term ultraviolet irradiation on transmission of three films.

weather-durable Mylar, and H-film is shown in figure 3. Figure 3(a) shows that the Tedlar-30 film had lost most of its transparency in the violet. The film became very brown in color and so the tests on this film were discontinued after 1071 hours. Electron damage tests on cells encapsulated with this plastic showed much damage to the film, which flaked off and turned brown after an exposure of 4×10^{16} electrons per square centimeter with 2.5-million-electron-volt electrons. This dose was about the same as that given the Tedlar 30 film earlier with no apparent effect. The lamination of the Tedlar 30 into a cell, however, may have affected the properties of the plastic in some undetermined way. Also, the energy of the electrons used to bombard the cell was slightly higher.

Figure 3(b) shows the transmission of the unirradiated weather-durable Mylar and the irradiated weather-durable Mylar at 3039 hours and at 5266 hours when the test was terminated. The film at this point was a light tan color, which is evidenced again by some loss of transmission in the violet end of the spectrum. The rate of darkening appeared to be faster initially. In fact, in the last 1000 hours of exposure time, the Mylar film did not darken appreciably.

Figure 3(c) shows the transmission of H-film at the beginning, at 3039 hours, and at 5266 hours. The transmission had changed only slightly in this length of time.

The results of the tests of the mechanical properties are shown in table III. Here the data for the unirradiated and the irradiated films are compared with each other and with the results from the early tests. The results of the short-

TABLE III. - COMPARISON OF MECHANICAL PROPERTIES OF FILMS FROM SHORT- AND LONG-TERM TESTS

[Short-term test (1003 hr): average temperature, 48° C; average pressure 3×10^{-6} torr; intensity, see table II. Long-term test: average temperature, 42.5° C; pressure range, 0.5×10^{-6} to 2×10^{-6} torr; intensity, equal to two-thirds integrated solar intensity below wavelengths of 300 mμ.]

Plastic	Elongation, percent (a)		Tensile strength at break, psi (b)		Total ultraviolet irradiation time, hr
	Unirradiated	Irradiated	Unirradiated	Irradiated	
Weather-durable Mylar	[c]120	69	19×10^3	22×10^3	5266
	120	(d)	24	10	1003
Tedlar 30	[c]270	130	11×10^3	13×10^3	1071
	270	54	11	8	1003
1-Mil H-film	210	110	25×10^3	29×10^3	5266
	110	78	21	20	1003

[a]Error: ±10 for percent >10, ±1 for percent <10.

[b]Error: $\pm2\times10^3$.

[c]Sample tore; values from start of short-term test.

[d]Sample too brittle to run.

and long-term tests are fairly consistent with each other. The difference in some of the values of percent elongation from one test to the other is probably due to differences in pulling rate; however, in each case, the unirradiated and the irradiated film are pulled under the same conditions, and the amount of change for each test is the significant datum. The most interesting result of these tests was the relative constancy of the H-film properties. The results indicated that this film is more than adequate for space use with respect to the effects of ultraviolet light on mechanical properties and transmission stability.

Second series. - The results of the tests run on almost all the additional samples obtained after the long-term tests had been initiated are summarized in table IV. The percent transmission for each sample is given at three representative wavelengths.

The poly (dimethylphenylene ether) and the poly (vinylidene fluoride) were tested because of their relatively good transmission from 350 to 750 millimicrons. The transmission diminished rapidly under irradiation, and the films turned a tan color. They were removed from the test chamber after 614 hours.

The silicone potting compound, which had been proved successful as a vehicle for zinc oxide thermal-control paints (ref. 9), also became darker with irradiation time, although after almost 1800 hours the film was still more transparent than 1/2-mil-thick H-film. The primer, tested originally to determine any effect that it may have had on the transmission of the silicone,

TABLE IV. - MECHANICAL PROPERTIES AND TRANSMISSION OF FILMS

[Test conditions: average temperature, 42.5° C; pressure range, 0.5×10^{-6} to 2×10^{-6} torr; intensity, equal to two-thirds integrated solar intensity below wavelengths of 300 mμ.]

Plastic	Total irradiation time, hr	Mechanical properties				Transmission, percent (c)									
		Elongation, percent (a)		Tensile strength at break, psi (b)		350 mμ		450 mμ		500 mμ		650 mμ		700 mμ	
		Unirradiated	Irradiated	Uniradiated	Irradiated	Unirradiated	Irradiated	Unirradiated	Irradiated	Unirradiated	Irradiated	Unirradiated	Irradiated	Unirradiated	Irradiated
Poly (2,6 Dimethylphenylene ether)	614	6	4	9×10^3	6×10^3	57	7	--	--	88	62	--	--	93	83
Poly (vinylidene fluoride)	614	(d)	10	7×10^3	6×10^3	70	28	--	--	75	52	--	--	86	77
Silicone primer, G. E. SS 4044	1782	(e)	(e)	(e)	(e)	91	77	--	--	93	90	98	96	--	--
Silicone potting compound, G. E. LTV 602	1782	(e)	(e)	(e)	(e)	91	23	--	--	93	76	98	93	--	--
H-film prepolymer															
Uncured	1997	[f]~20	1	[f]9×10^3	2×10^3	(g)	(g)	84	39	89	65	93	86	--	--
H_1, cured 2 min at 400° C	1997	17	12	11×10^3	12×10^3	(i)	(i)	20	19	67	65	92	92	--	--
H_2, cured 100 min at 200° C	1997	19	[h]8	14×10^3	[h]12×10^3	(i)	(i)	20	19	76	72	93	92	--	--
H_3, cured 16 hr at 150° C	1997	4	6	11×10^3	12×10^3	(i)	(i)	24	22	73	67	93	92	--	--
1/2-Mil H-film	(j)	61	--	19×10^3	--	(i)	(i)	10	--	68	--	94	--	--	--
1-Mil H-film	5266	210	110	25×10^3	29×10^3	--	--	(k)	(k)	47	43	90	90	--	--

[a]Error: ±10 for percent >10, ±1 for percent <10.
[b]Error: $\pm2\times10^3$.
[c]Error: ±1 percent for wavelengths <700 mμ, ±3 percent at 700 mμ.
[d]Two samples tested; both tore.
[e]Coating painted on glass slide.
[f]Original film embrittled on aging; value is for newly cast film.
[g]Film completely absorbing below 380 mμ.
[h]Estimated; sample too small for measurement.
[i]Film completely absorbing below 400 mμ.
[j]Sample not irradiated.
[k]Film completely absorbing below 450 mμ.

turned out to be considerably more resistant to ultraviolet damage than the silicone paint itself. After the same exposure time, the transmission had decreased only slightly; this certainly makes it worthy of further study. No mechanical properties could be measured on these films since they were painted on glass plates.

The rest of the films will be treated as a group; these are the H-film prepolymer, prepared by casting a solution of Pyre M-L varnish in dimethylformamide, and some of this same film cured at various time-temperature combinations. All these films are approximately 1/2-mil thick, and therefore the properties of both 1/2- and 1-mil H-film are included for comparison.

The prepolymer is of interest because it transmits light of shorter wavelengths than either 1/2- or 1-mil H-film and becomes completely absorbing below 380 millimicrons, whereas the others are completely absorbing below 400 and 450 millimicrons, respectively. This property could increase the performance of a solar cell, other things being equal, since cadmium sulfide films have considerable response below these wavelengths. Under ultraviolet irradiation, however, the uncured film gradually darkened and after almost 2000 hours approached 1/2-mil H-film in its transmission. In addition, the film became extremely difficult to handle after irradiation, and its mechanical properties were very poor.

The three samples of the prepolymer which were cured, H_1, H_2, and H_3, became obviously yellower in color. Their transmission was about the same as that of 1/2-mil H-film except in the low wavelength cutoff region. The transmission remained relatively constant throughout the radiation test. The mechanical properties of the originals were generally poorer than those of 1/2-mil H-film and worsened with irradiation.

The composite film of the prepolymer H-film cast on weather-durable Mylar was irradiated for a total of 762 hours. Since the thickness of the composite was $1\frac{1}{2}$ mils and the original Mylar was 1 mil thick, the prepolymer layer was about 1/2 mil. The transmission of the film (better than 1/2-mil H-film) did not change in this time, and the percent elongation remained good. The original composite separated into two films, but the irradiated film remained intact. More careful techniques in applying the prepolymer have produced better adherence and a more even and thinner coat.

The results of this study seem to indicate that, of the films tested, H-film is superior with regard to ultraviolet damage. (The silicone primer is being tested further.) The fact to be remembered, however, is that these films are of interest as cell encapsulants, and the performance of a cell will depend not only on how its protective coating will stand up in the space environment but also on how the coating affects the performance of the cell itself.

Comparison of figures 3(b) and (c) (p. 9) shows that, initially, weather-durable Mylar will allow more of the solar spectrum to reach the cell than the H-film. The transmission of the solar spectrum to the cell can be improved slightly for H-film encapsulated cells by using thinner films, but thicknesses less than 1/2 mil have not been manufacturered. This loss of light to the cell results in an average decrease in cell efficiency of about 15 percent. With time, the transmission of the weather-durable Mylar will drop and gradually approach (but always remain higher than) that for H-film, so that Mylar encapsulated cells will tend to drop in efficiency while those in H-film will tend to remain the same. Mylar-coated cells would be favored as a power source where the brittleness of the Mylar due to ultraviolet damage would not be important since the efficiency of these cells is higher because of the higher transmission of the film at lower wavelengths. Many other considerations are involved in cell performance, however. A continuing search is being made for coatings that will improve cell properties in one area without deteriorating them in another or perhaps even better other properties also.

CONCLUSIONS

Irradiation of selected plastic films in vacuum by ultraviolet light of intensity 0.67 to 1 times the integrated solar intensity below 3000 angstroms shows that, with respect to transmission stability and retention of useful mechanical properties (particularly percent elongation), H-film, a polyimide film, is superior. Weather-durable Mylar, however, although it does darken and become somewhat more brittle, may be as useful in some applications. This usefulness may be true in the particular area of interest, encapsulants for thin-film solar cells.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, May 17, 1965.

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NASA-Langley, 1965 E-2990

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